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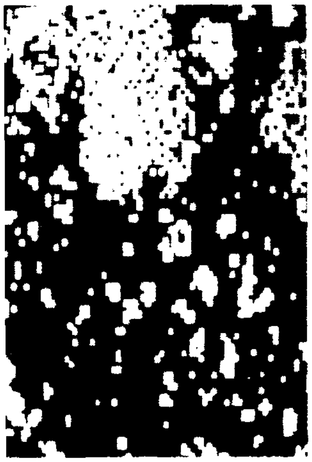

STRUCTURES LABORATORY

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TECHNICAL REPORT SL-92-19

PETROGRAPHIC TECHNIQUES APPLIED TO CEMENT-SOLIDIFIED HAZARDOUS WASTE

by

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US ARMY ENGINEER WATERWAYS EXPERIMENT STATION

August 1992

Final Report

Prepared for US Environmental Protection Agency
Risk Reduction Engineering Laboratory
Cincinnati, Ohio 45268

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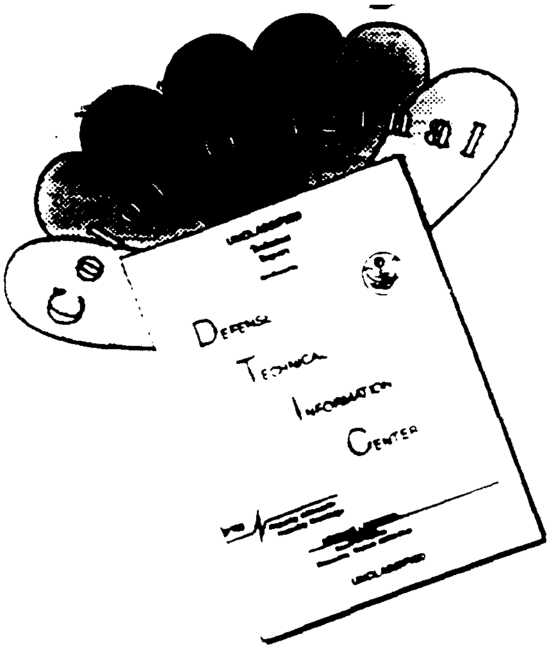

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REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1. AGENCY USE ONLY (*Leave blank*)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	August 1992	Final report

4. TITLE AND SUBTITLE

Petrographic Techniques Applied to Cement-Solidified Hazardous Waste

5. FUNDING NUMBERS

6. AUTHOR(S)

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7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

USAE Waterways Experiment Station
Structures Laboratory
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

8. PERFORMING ORGANIZATION REPORT NUMBER

Technical Report SL-92-19

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

Risk Reduction Engineering Laboratory
US Environmental Protection Agency
Cincinnati, OH 45268

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

Available from National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution is unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT (*Maximum 200 words*)

Twelve techniques used routinely in laboratory investigations of portland-cement concretes and grouts were applied to three cement-solidified wasteforms from an EPA Superfund site. The objective was to determine if the presence of the wastes made these solidified materials fundamentally different from conventional grouts. Other objectives were to determine if techniques used routinely in concrete technology are applicable to such wasteforms and to identify what unique and useful information they can provide for determining the likelihood for durability of the wasteform.

Petrographic examination of thin sections of wasteforms prepared with fluorescent epoxy resin proved to be useful for determining effectiveness of waste dispersion in the cementing medium and distribution of cracks. Scanning electron microscopy coupled with high-resolution X-ray mapping revealed details of microstructure and distribution of important ions. X-ray powder diffraction

(Continued)

14. SUBJECT TERMS			15. NUMBER OF PAGES
Cement microscopy	Image analysis	(Continued)	29
Concrete petrography	Pulse velocity		16. PRICE CODE
Grout	Scanning electron microscopy		

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED		

13. ABSTRACT (Continued).

confirmed the formation of the usual hydrated phases of portland cement and pozzolans. Pulse velocity measurements indicated absence of serious degradation over an 18-month period of time.

The petrographic and nondestructive analytical techniques common in concrete technology and applied to wasteform grouts in this study can be applied to these cement-solidified wastes. These techniques indicate if the materials have stable phase composition, if their microstructures demonstrate uniform waste dispersal, and if mass or structural integrity is sufficient so that large blocks of these materials are unlikely to disintegrate in place.

14. SUBJECT TERMS (Continued).

Solidification
Solidified waste
Stabilization
Wasteform

PREFACE

This report summarizes major findings from a 2-year research effort accomplished in the Concrete Technology Division (CTD), Structures Laboratory (SL), US Army Engineer Waterways Experiment Station (WES), under contract to the Risk Reduction Engineering Laboratory, US Environmental Protection Agency (EPA), Cincinnati, OH. Dr. Walter E. Grube, Jr., was the Technical Monitor of this work for EPA.

Members of the staff of CTD who accomplished this work included: Dr. Lillian D. Wakeley, Messrs. G. Sam Wong and J. Pete Burkes, and Mrs. Judy C. Tom, with assistance from Messrs. Charles L. White, Michael I. Hammons, and Mrs. Linda S. Mayfield. Mr. Wong and Dr. Wakeley also participated in the field examination of the large blocks cast and stored at the subject EPA Superfund Site, for which Mr. Wong was project leader for WES. The work was performed under the general supervision of Mr. Kenneth L. Saucier, Chief, CTD, and Mr. Bryant Mather, Director, SL. This report was prepared by Dr. Wakeley with assistance from Messrs. Wong and Burkes.

At the time of publication of this report, Director of WES was Dr. Robert W. Whalin. Commander and Deputy Director was COL Leonard G. Hassell, EN.

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PETROGRAPHIC TECHNIQUES APPLIED TO CEMENT-SOLIDIFIED HAZARDOUS WASTE

PART I: INTRODUCTION

1. Cements and other materials common in concrete construction are used widely for solidification and stabilization (S/S) of hazardous wastes. There are many good reasons for using cements in this way: They form a solid at room temperature, are inexpensive to buy and transport, can be handled and mixed with off-the-shelf equipment, and are common all over the world. Because cements are so familiar, the technology of their use seems simple, and this, too, is appealing for waste solidification.

2. Successfully stabilizing, or at least solidifying, a waste is far removed from grouting a foundation, or the ultimate pejorative, "just making concrete."* A solidified waste is neither a structural material nor a space-filling slurry, so it is neither concrete nor grout. Instead, the cementitious solids of a solidified waste bind the waste physically and may bind it chemically, allowing it to pass whatever performance tests are required for transporting and disposal. The cement content is kept as low as possible, and the waste loading as high as possible, to reduce materials costs and avoid producing unmanageable quantities of solid product.

3. A largely separate family of standard test methods has evolved for solidified wastes, because this is an entirely separate use for cements. Some of these methods refer to or were based on standard methods from concrete technology. For example, ASTM D 4842, "Standard Test Method for Determining the Resistance of Solid Wastes to Freezing and Thawing," refers to ASTM C 305, "Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars"; and produces data on mass loss through a fixed number of cycles of freezing and thawing, as does the much older standard for concrete, ASTM C 666 (American Society for Testing and Materials 1991). Although it is obvious

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* Concrete is a cement-based structural material which is commonly composed of up to 75% aggregates, by volume. In other words, cement is to concrete as flour is to fruitcake. Grout is a slurry of cement and water, with or without other components, and is not a structural material.

that some standard or routine procedures of concrete technology apply to solidified wastes, it is not obvious which ones <u>do</u> apply, nor how they apply.

Objectives

4. We used twelve techniques used routinely in investigations of concretes to study three cement-solidified wasteforms. The objectives were: (a) determine if these techniques are applicable and appropriate to cement-solidified wastes; (b) determine if the solidification technology used for these particular wastes was effective in dispersing the waste throughout the wasteform monoliths; (c) determine what types of evidence indicate whether the wastes were chemically bound or only physically contained within the cementitious matrix; and (d) decide if any of the techniques investigated can be recommended for routine or standard practice to indicate likelihood of wasteform durability. Because this study was conducted by concrete technologists, we asked ourselves an additional question: Did the presence of the waste cause the cementitious matrix to form a solid that is fundamentally different from what it would have formed in a grout or concrete (i.e., is this the moral equivalent of concrete)?

Wastes and Solidification Technology

5. The US Environmental Protection Agency (EPA) selected a solidification technology developed by Soliditech, Inc., of Houston, TX, to be demonstrated under the Superfund Innovative Technology Evaluation (SITE) program. Rather than dealing with research and development of waste-treatment technologies, the SITE program uses commercially available systems on Superfund-listed sites to demonstrate the application of these systems to real problems. The Soliditech system consisted of cement-based materials with proprietary additives and water and the mixing process during which appropriate components and proportions were selected by Soliditech employees. Monoliths (approximately 1 m^3 each) of the treated wastes were cast at the Superfund site during the Soliditech demonstration, as were cylindrical samples for offsite testing and monitoring.

6. The wastes were remnants of an oil-recycling facility, and consisted of contaminated soil, filter cake, and a mixture of filter cake and oily sludge. Principal contaminants in the oily wastes were lead, arsenic, toluene, and PCBs. Clean sand was used to replace waste for control specimens for full-sized monoliths and cylinders. A description of the Soliditech process, proportions of S/S materials, the field activities, and analyses of the wastes are available in an EPA Applications Analysis Report (US Environmental Protection Agency Risk Reduction Engineering Laboratory (USEPARREL) 1990).

PART II: FIELD TECHNIQUES

7. Six months after the monoliths were cast, we observed the monoliths at the Superfund demonstration site. During that site visit, monoliths were described following standard practices for concrete condition surveys (ACI 201.R) (American Concrete Institute (ACI) 1990). Size, spacing, and distribution of cracks were noted as was evidence of inhomogeneous mixing or surface alteration. Other features noted during these inspections were spalling, surface features, changes in color, and efflorescence. Crack monitoring gages were installed and monitored, as described in ASTM C 426, "Test Method for Drying Shrinkage of Concrete Block" (ASTM 1991), modified to accommodate field conditions. Site visits were repeated at 1 year and 18 months. Field observations and study techniques are presented in a WES report (Wakeley and Wong in preparation).

PART III: LABORATORY TECHNIQUES

8. Other laboratories (Radian Corporation; PRC Environmental Management, Inc.) performed the primary evaluation of the wastes and treated wastes of this SITE demonstration. They approached the study from their experience with solidified wastes, and performed appropriate physical tests, such as bulk density, water content, permeability, wet/dry weathering, and loss on ignition, and extensive leach testing and chemical analyses. Results of this work are presented in an EPA Technology Evaluation Report (USEPARREL 1990).

9. For the additional studies at WES, we selected techniques familiar to research on cement-based concretes and grouts and which we judged to have the most potential to provide information useful to evaluating cement-solidified wastes. These techniques fit generally into three categories: nondestructive techniques, techniques for studying morphology and texture at moderate magnification, and techniques to study microstructure and chemical components.

Nondestructive Techniques

10. Techniques considered nondestructive were applied to whole cylinders of field-cast solidified waste and control mixtures (Figure 1). Visual examination, X-ray radiography, and ultrasonic wave velocity were the principal nondestructive techniques applied. During visual examination, features such as color, cracks, distinct particles or inhomogeneities, and evidence of segregation visible on the perimeter of each specimen were documented. Guidance for visual examination is given in ASTM C 856, "Standard Practice for Petrographic Examination of Hardened Concrete" (ASTM 1991). Features observed were correlated to images from X-ray radiography, which was performed to determine its utility as a technique for revealing inhomogeneity. Air voids and regions of solids that are less dense than the cementitious matrix cause darker areas in the negatives (Figure 2). Radiographs were recorded using 300 KV and 10 ma for 105 sec.

11. The principal quantitative technique of nondestructive evaluation (NDE) applied to solidified wastes in this study was pulse

velocity. The velocity of a mechanical pulse through a material can be used comparatively to indicate change or constancy of mechanical properties of the material. This technique is described elsewhere (Malhotra 1976; Thornton and Alexander 1987; and ACI 1988). An initial pulse velocity measurement is the baseline value to which you compare pulse velocity values taken at later times. If the value decreases markedly with time, this indicates probable physical deterioration which can be confirmed by other tests. This value can be measured within about 1.5 m/sec for a typical velocity of 4,500 m/sec, making it a very precise tool for detecting changes over time.

12. This NDE technique is appropriate to solidified wastes because it is nondestructive and permits retest of the same specimen many times during long-term monitoring. Also, it is particularly well suited to cement-based solids, because the properties of hardened cements change with time and independently with exposure to certain physical or chemical conditions. In this study, the initial values were read for field-cast cylinders at 6 months, with subsequent monitoring at 12 and 18 months. Data from these tests are shown in Figure 3.

Morphology and Texture

13. Petrographic thin sections were prepared for optical microscopy using techniques similar to those widely practiced for geologic materials (Bloss 1961). We modified the standard practices of slabbing, grinding, and polishing to minimize alteration of either the cement matrix, which reacts with water, or the oily waste, which reacts with or is soluble in organic solvents. We expected to be able to identify the hydrated cement and fly ash components of the solidification matrix just as we do for any cementitious material, unless the matrix had reacted extensively with the wastes. We also anticipated that the distribution of oily particles in the matrix would be obvious by fluorescence in ultraviolet light.

14. To prepare acceptable thin sections (for which the sample is thinned to less than 0.1 mm thick, permitting light to pass through and giving characteristic optical signatures for each component), it was necessary first to impregnate each sample with epoxy resin. When we determined that the oily material in the wastes did not fluoresce in UV light, we prepared a second set

of thin sections, using an epoxy to which a fluorescent dye had been added. This permitted direct observation of the distribution of waste particles (described below) and of pores and cracks. Details of sample preparation and petrographic observations are in a WES report (Wakeley and Wong in preparation).

15. Study of the thin sections revealed a cementitious matrix with no unique features attributable to interaction between cement and waste (Figure 4). The fluorescent thin sections showed fairly uniform distribution of waste particles in most specimens. The waste particles were revealed to be more porous than the cementitious matrix, creating brightly fluorescing areas when observed in UV light. The waste was revealed to be distributed as compound particles, resembling clumps of silt agglomerated by their oil coating (Figure 5).

16. Slabs of each solidified waste were prepared for optical examination at lower magnification in reflected light. Again, samples first were impregnated with an appropriate epoxy resin and examined as described in ASTM C 457, "Practice for Microscopical Determination of Air-Void Content On a Parameters of the Air-Void System in Hardened Concrete" (ASTM 1991). Percentages of each component were determined by point counts, with particles identified in these categories: waste particles larger than 1.0 mm, waste particles 1.0 mm or smaller, cementitious matrix, air void, and aggregate (sand particles in the control specimens). These counts were intended to indicate differences in waste distribution or agglomeration and porosity for the three waste types, for comparison to other properties.

Microstructure, Elemental and Phase Composition

17. Phase composition of the inorganic portion of all solidified wastes was determined by X-ray powder diffraction (XRD). The oily components were extracted during several days of treatment in Soxhlet apparatus with a suitable solvent. Although some carbon residue remained, the resulting inorganic solids were successfully prepared as powders for XRD.

18. We determined chemical composition by energy-dispersive X-ray microanalysis (EDX), performed in conjunction with scanning electron microscopy (SEM). We were not interested in representative chemical

compositions of the wastes or the matrix, these having been determined previously (USEPARREL 1990). Instead, we used SEM to locate certain areas or particle types and EDX for elemental composition of those areas. Carbon (elemental C) was assumed to represent the oil component of the waste materials. So we used high-resolution X-ray mapping to locate concentrations of carbon and of other key elements (such as Ca and Si), and determine co-locations among them (Figure 6).

PART IV: RESULTS

19. We do not present the data from all these analyses in this paper. We offer our opinion of which techniques show the most promise as tools for monitoring the solidified/stabilized waste in this study. Wastes vary so much that we do not consider these data to be typical of anything, nor can we be sure that these techniques will be appropriate to other hazardous materials solidified by other systems.

20. We were encouraged by the consistent data derived from petrographic observations, high-resolution X-ray mapping, and pulse velocity. These techniques allowed us to relate various features to original composition, and determine whether the wasteform was stable or changing with time. Petrographic observations, including field data, indicated differences in size and distribution of waste particles and in the tendency to crack. These differences could be related to cement content, ratio of water to cementitious materials, and to a lesser extent to waste loading.

21. The content of each mixture reported (USEPARREL 1990) as "waste material" was misleading, as we discovered in the thin sections, during solvent extraction, and by X-ray mapping. Each reported percentage of "waste material" actually included a large portion of chemically inert mineral matter, particularly for contaminated soil from offsite. These "wastes" behaved more as aggregate particles than as wastes, allowing very low cement contents in the total formulation. It was not surprising to us, then, that the solidified contaminated soil behaved similarly to a conventional sanded grout, having in common with grout a low cement content and large content of fine-aggregate equivalent (the "waste"). For a different waste, the formulation of materials chosen by Soliditech would have produced a solid with totally different properties.

22. The X-ray mapping showed clearly that carbon, representing the oil, was positively associated with silicon, without associated calcium. The oil is clumped with siliceous particles that make up most of the mass of the waste, and is not dispersed through the matrix (of calcium silicates and aluminates -- cement and fly ash) or combined with it to form new phases. Both the easily seen and the microscopic oily particles were physically encapsulated by the matrix, and remained fixed throughout the two-year period

of our observations. Carefully interpreted petrography and EDX told us much that was useful about these solidified wastes.

23. Pulse velocity measurements correlated well with our observations and interpretations of microstructure and composition. They indicated no change or very little change with time, as another line of evidence that the matrix component remained in a stably hydrated condition from six months (the time of our initial observations, after the matrix had had plenty of time to hydrate) to a year and a half. The control specimens, of matrix plus clean sand, showed a predictable increase in pulse velocity, as expected for a high-quality sanded grout. By this technique, we would have detected any notable deterioration of these specimens. Because it is nondestructive, and capable of providing both an initial signature and a long-term profile of change or constancy, measurements of pulse velocity show promise as a screening tool. It can tell you when your wasteform has a problem, so that other techniques can be applied to interpret and address that problem.

24. Radiography did not provide uniquely useful data in this study. However, we had the least experience with this tool, and the equipment was older and less sophisticated and thus not comparable to the instrumentation we used for the other analyses. None of these tools alone answered all of our questions. But a thoughtful application of petrographic observations and pulse velocity, particularly with X-ray mapping, looked promising.

PART V: DISCUSSION

25. To interpret the results of analyses of cement-based materials, recall that their properties are dependent on both environment and time. The treated waste does not get hard because the cement dries, as is commonly assumed. If all that was required was drying, you could do the job just as well by mixing the waste with wet mud and letting it dry. A grout hardens because cement reacts chemically with water to form new, strength-giving compounds. Adding any waste to that grout introduces new chemicals, with which come the risk that the compounds now forming will give no strength; or they will be unstable in water; or that the strength-giving phases never will form and the grout never will set; or worse, that they will un-form, and the material will disintegrate spontaneously in the future. A solid monolith of treated waste -- or wasteform grout -- may not be solid a year from now, because of delayed or cumulative chemical interactions between waste and matrix.

26. Long-term performance of a solidified waste becomes important if the waste is solidified months to years before it is transported to a licensed facility for permanent disposal, a situation which seems to be increasingly common. The blocks solidified during this SITE demonstration, for example, remained where they were cast for more than three years, awaiting transportation and disposal elsewhere. A successful solidification technology, then, is one the products of which pass all required tests soon after casting and remain solid and stable enough to be transported offsite at some unspecified later time.

27. An environmental engineer and a materials scientist may look at the monoliths in this study in fundamentally different ways. The former is likely to see treated waste, and the latter to see grout with contaminants in it. Both want the solid to have the minimum cement content required to do the job, for a combination of reasons including keeping costs reasonable, minimizing the volume of treated waste needing disposal, and minimizing heat generated during hydration thus to reduce thermal stresses and cracking. As a treated waste, the material needs to pass leach tests, have a compressive strength of at least 0.345 MPa (500 psi), and survive 12 cycles of freezing and thawing with at least 70 percent of its original mass intact (ASTM D 4842 (ASTM

1991)). The fact that these properties are acceptable for treated wastes shows the enormous difference between waste treatment and other technologies using cements.

28. Practitioners in the field of concrete technology are accustomed to worrying about long-term performance of cement-based solids. This may be the subject area on which we have the most to offer to practitioners of solidification/stabilization. Analytical techniques borrowed from materials science and concrete technology can help answer these questions: If the treated-waste blocks remain onsite for three or four years, can they still be picked up with a forklift, loaded three-deep on a flatbed truck, transported, offloaded, and buried, without being squashed or falling apart? Or will components of the blocks react with water, form new and unstable phases, fracture readily as they freeze and thaw, and by various chemical and physical changes degrade during their indefinite years of temporary storage? Will they unharden in their storage environment?

29. If you want techniques for determining the likelihood of long-term solidification or durability of a wasteform, nondestructive evaluation can be conducted initially and repeated fairly easily at intervals to indicate whether the wasteform is stable or changing. Phase composition may be at least as important to wasteform durability as total chemical composition: The crystalline form accommodating a particular cation determines its stability. X-ray diffraction, used commonly in concrete forensics and petrography, is a technique capable of distinguishing among crystalline forms with a common chemical composition. The total chemical composition of a solid may remain nearly constant as it undergoes phase changes and submicroscopic shifts that drastically alter its strength or leach resistance.

30. The petrographic and nondestructive analytical techniques used routinely in concrete technology can be applied to some cement-solidified wastes to indicate if a material has a stable phase composition, if its microstructure demonstrates uniform waste dispersal, and if mass or structural integrity is sufficient so that it will not disintegrate in place. These techniques can be used for quality assurance monitoring of stabilized wastes, or applied in a forensic sense to materials that have failed, to identify the chemical and physical causes of deterioration so that the problem can be corrected during reformulation.

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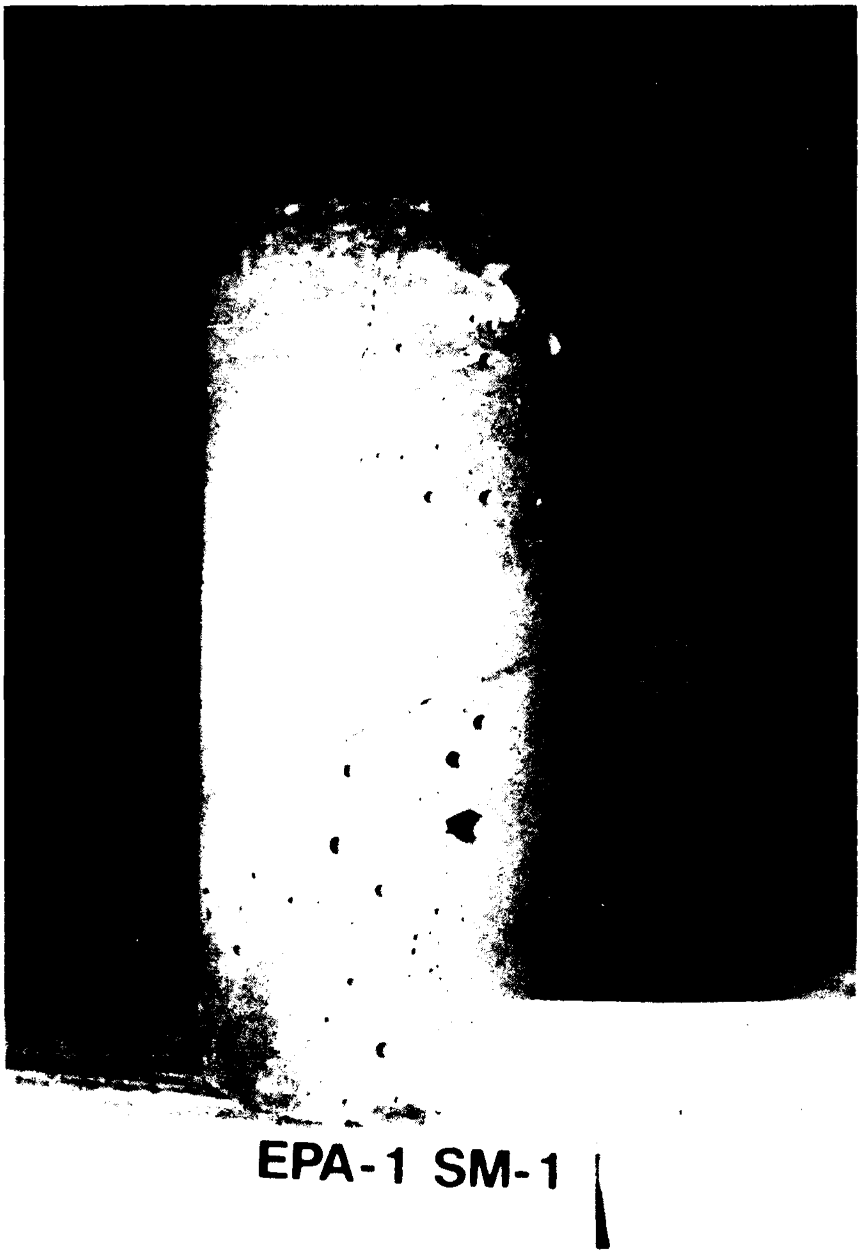


Figure 1. Cylinder of solidified filter cake. Diagonal line on circumference shows location of seam in cardboard sample mold; surface voids represent air trapped during casting

Figure 2. X-ray radiograph of representative cemented-waste cylinder (height=145 mm). Low density areas are voids

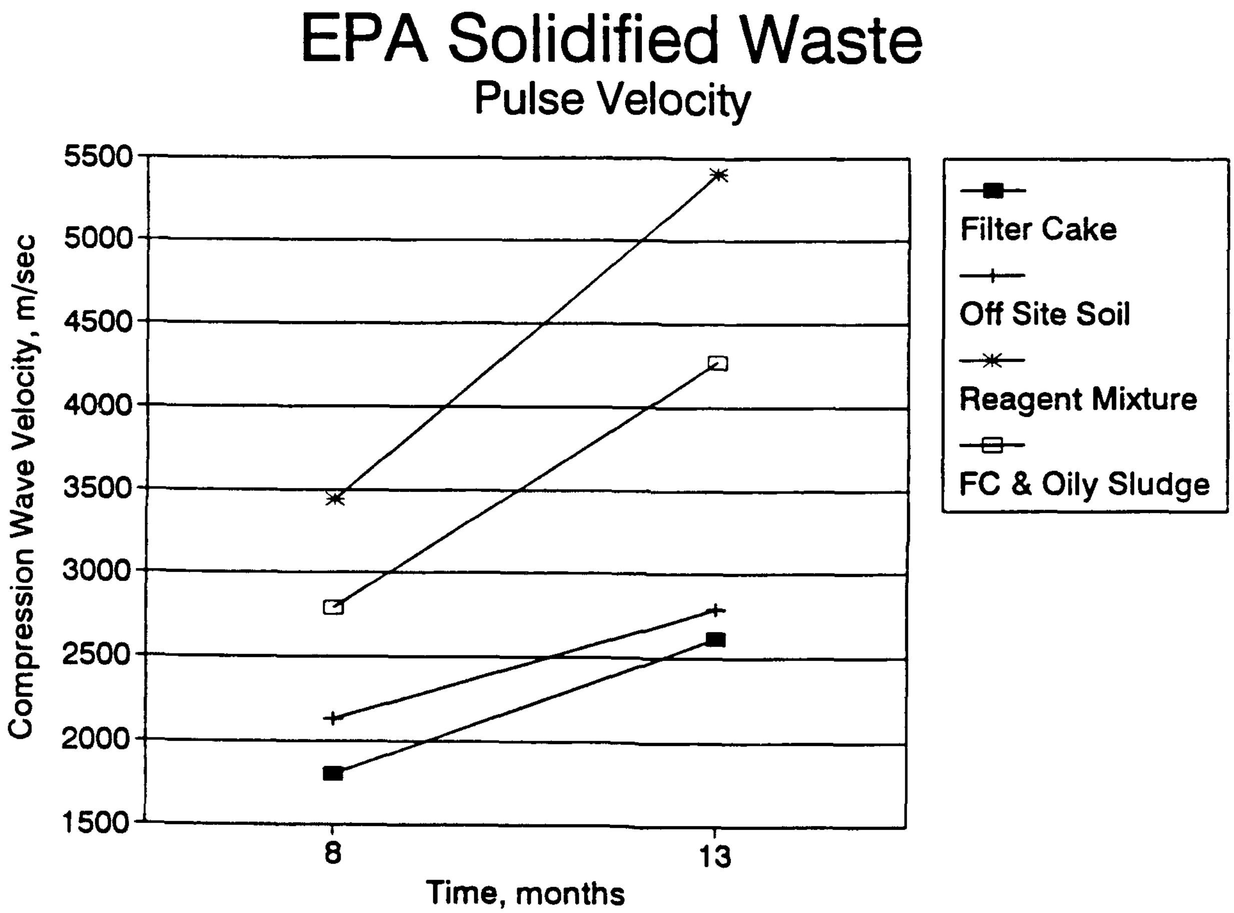


Figure 3. Data from pulse velocity measurements of cylinders of three types of solidified waste and control mixture at 8 and 13 months age

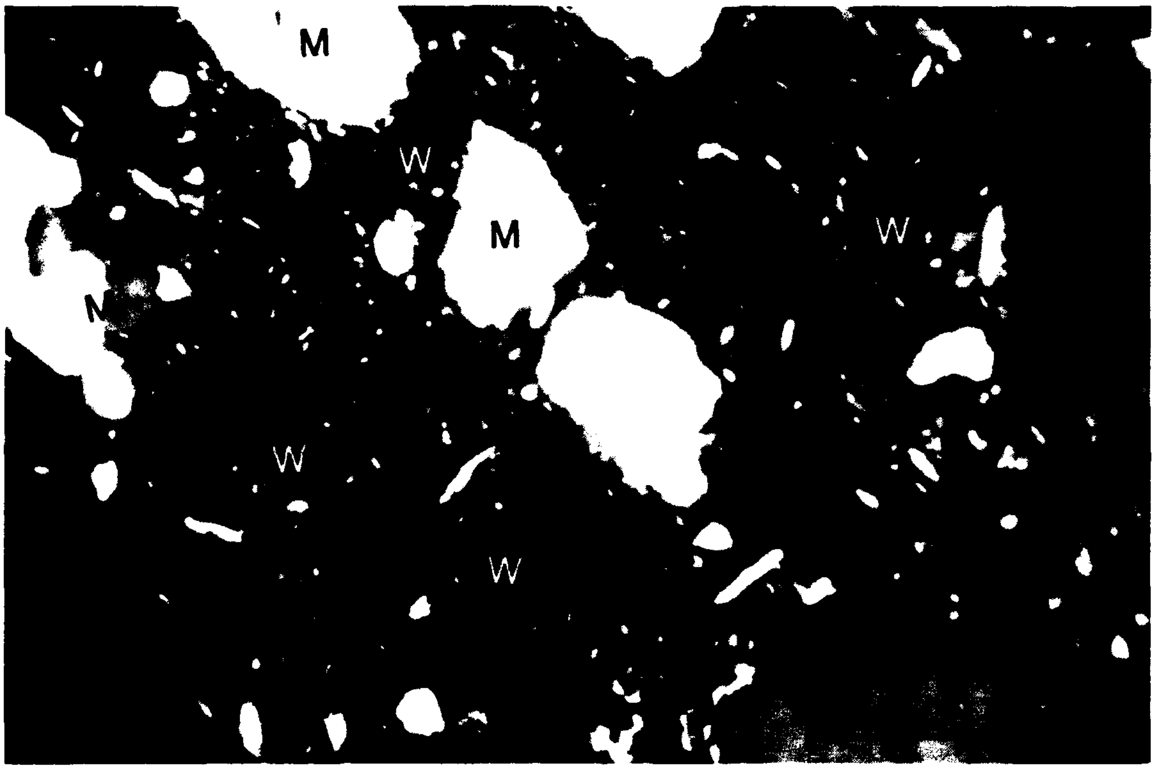


Figure 4. Thin section of sample of cemented filter cake plus oily sludge, showing dispersed oily particles of waste (w) and mineral matter (m); viewed in cross-polarized light

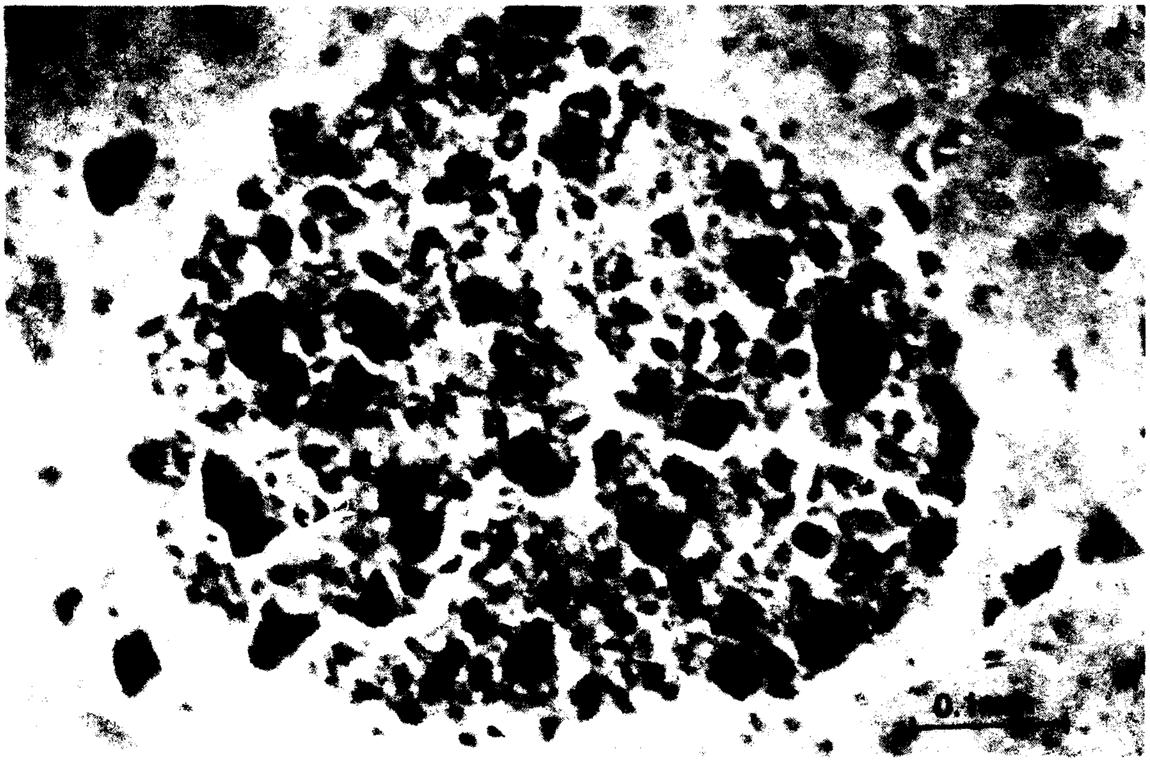
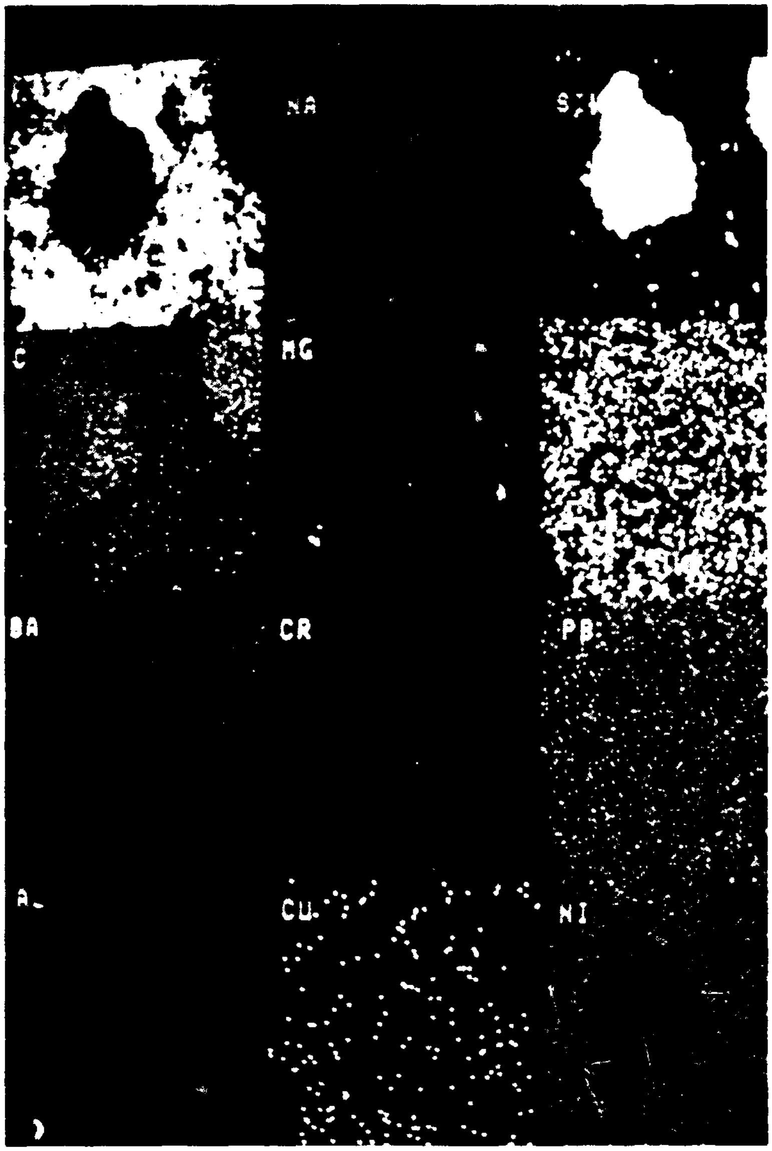
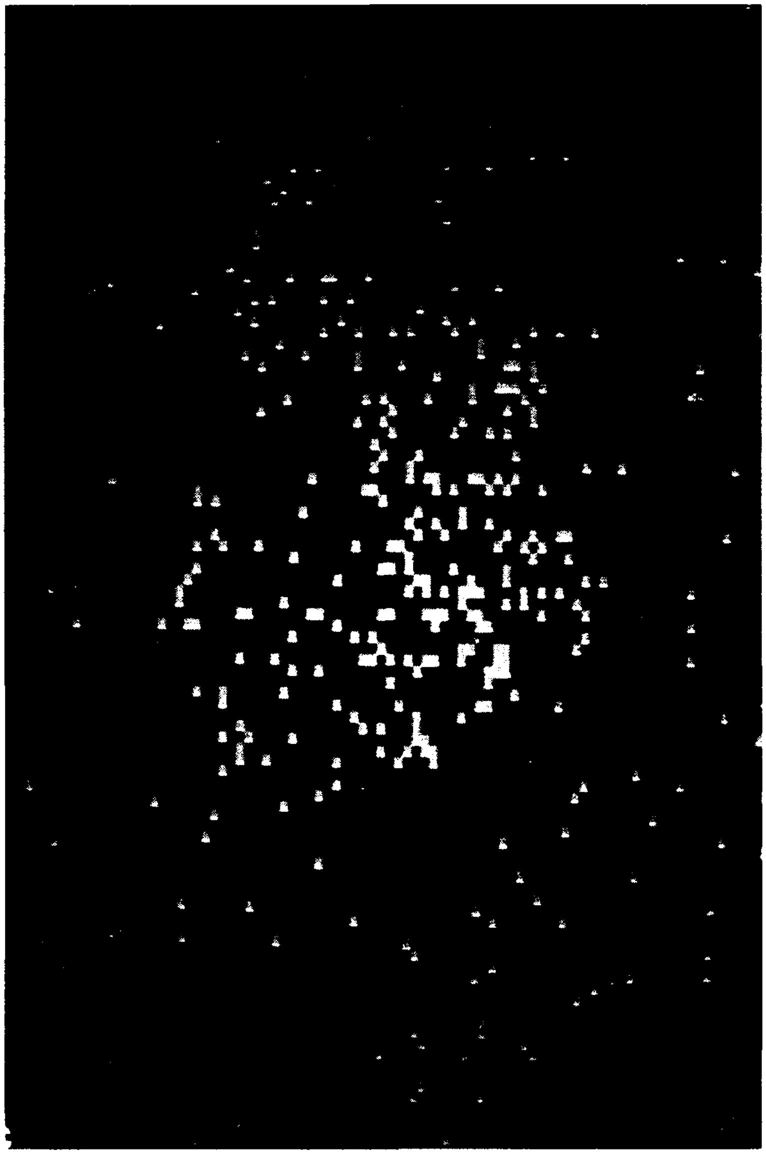


Figure 5. Thin section prepared after impregnation with fluorescent epoxy of sample of cemented contaminated soil from offsite, showing porous oil-contaminated soil agglomerate, viewed in ultra-violet light



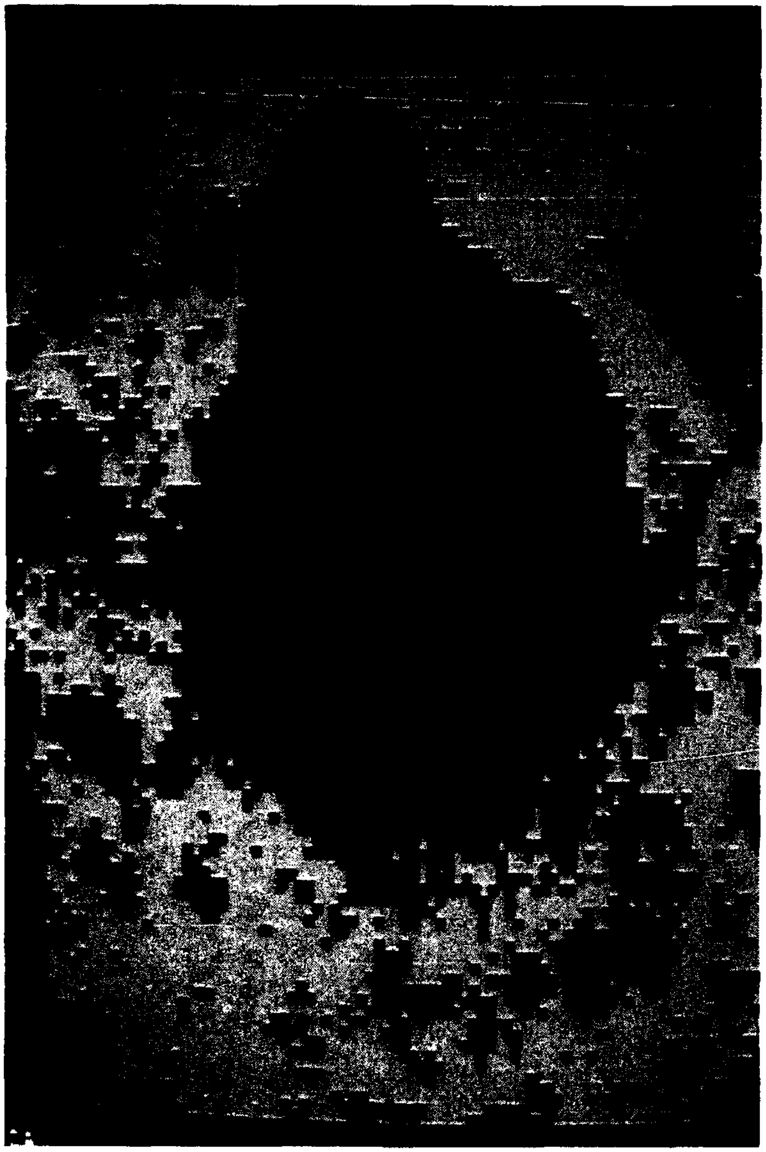
a. Element distribution maps showing (across from upper left) calcium, sodium, silicon, carbon, magnesium, zinc, barium, chromium, lead, aluminum, copper, and nickel

Figure 6. Element-distribution maps on and around an oil-contaminated soil agglomerate from offsite, image analyses of EDX data. Soil particle is approximately centered in image, and is 1.4 mm across (Sheet 1 of 5)



b. Distribution of carbon, showing its concentration in contaminated soil particle

Figure 6. (Sheet 2 of 5)



c. Distribution of calcium, showing its concentration outside soil particle in cementitious matrix

Figure 6. (Sheet 3 of 5)

d. Distribution of silicon, showing its concentration in contaminated soil particle

Figure 6. (Sheet 4 of 5)

e. Composite distribution of carbon (green), silicon (blue), and calcium (yellow), showing positive association of carbon and silicon (quartz and clay minerals) in oil-contaminated soil particle

Figure 6. (Sheet 5 of 5)